RESEARCH ARTICLE

Effect of Fiber Modification on Pineapple Leaf Fiber's Mechanical Properties and Interfacial Adhesion

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ABSTRACT: Due to their key benefits, which include having a moderate strength and stiffness, being inexpensive, and being a material that is good for the environment, degradable, and renewable, natural fibers are seen as having potential application as reinforcing agents in polymer composite materials. Because of their natural hydrophilicity, they are prone to absorbing moisture, which can weaken or plasticize the adhesion of the fibers to the surrounding matrix and affect how well composite materials operate in ambient humidity, especially at high temperatures. The aim of this study was to examine the effects of treatments on the mechanical (tensile), morphological, and structural characteristics of pineapple leaf fibers using sodium hydroxide (alkali), 3-aminopropyltriethoxysilane (3-APS, silane), and combinations of the two. The effectiveness of the alkali and saline treatments in eliminating impurities from the fiber surfaces was demonstrated by scanning electron microscopy (SEM) examination. According to the morphological study of treated PALF by SEM, silane-treated fibers have less impurities, lignin, and hemicelluloses eliminated than those by other chemical treatments. The tensile strength of silane-treated PALF is greater than that of untreated, alkaline, and NaOH-silane-treated PALF. A single-fiber fragmentation test and data reduction technique were utilized to assess the interface quality of untreated, NaOH, silane, and NaOH-silane treated PALFs-epoxy composites, as well as the apparent interfacial shear strength (IFSS). It was found that treated samples greatly increase IFSS, indicating an increased level of adhesion. The silane-treated fibers exhibited the highest interfacial stress strength, whereas the PALF treated with alkali and silane had better interfacial stress strength, according to the droplet test. It is envisaged that fiber treatments will aid in the creation of high performance PALF reinforced polymer composites for industrial applications.

KEYWORDS: Epoxy resin, fiber-matrix interfacial adhesion, single-fiber droplet test, pineapple leaf fiber (PALF), and surface treatments

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INTRODUCTION

There is an increasing interest in materials science to employ natural fibers (NFs) as reinforcement of polymer composites. The density of natural fibers is low (1.15-1.50 g/cm^3) vs E-glass has excellent specific mechanical characteristics and a density of 2.4 g/cm^3 [1]. Additionally, natural fibers are widely accessible and cost around one-sixth as much as glass fibers (3.25 $/kg). The most important components in a variety of industrial purposes, such as textile, paper, packaging, and building materials, are natural fibers [2-4]. Because natural fibers are renewable and biodegradable, products made from them are also environmentally friendly. Natural fibers are superior to synthetic fibers in several areas, including having high flexural strength, low density, strong thermal insulation [5, 6] and outstanding acoustic properties [7]. Applications involving building materials are strongly advised to take use of these advantages, which are currently being used in a range of applications [8]. Natural fibers are made of cellulose, hemicellulose, lignin, pectins, waxes, and water soluble substances [9]. However, aging, the retting process, and environmental factors also have an impact on the chemical makeup and physical properties [10]. Numerous fiber plants, such as pineapple, kenaf, coir, abaca, sisal, cotton, jute, bamboo, banana, palmyra, talipot, hemp, and flex, can be utilized as raw materials in a variety of businesses. Easily accessible PALF garbage from South-east Asian nations is used to create fibers.

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Pineapple-leaf fiber (PALF) is one of the most important plant-based fibers for composite materials due to its moderate specific strength and stiffness compared to other natural fibers. They are therefore in direct competition with glass fibers for use as a reinforcing component in composite applications [11]. Natural fiber polymer composites having a minimal influence on the environment, and depending on the polymer matrix, they may be referred to as eco- or green composites [12,13]. In recent years, some well-known lignocellulosic fibers have undergone substantial research and have been used as reinforcement in polymer composites. These investigations and application initiatives were covered in journal papers, book chapters, and review articles [14-16]. In particular, it was noted that the tensile strength changes with the inverse of the diameter for sisal, ramie, curaua, jute, bamboo, coir, piassava, and buriti fibers. The potential use of PALFs as reinforcing materials in a range of polymers, including epoxy resin [17,18], polyester resin [19,20], polypropylene [21,22], polyethylene [23,24], polylactic acid [25,26], and natural rubber [27, 28], has been highlighted in a number of studies over the past few years. In PALF reinforced polymers, it is well known that adhesion between the PALFs and the matrix, a composite material with weak interfaces and low strength and stiffness, has a substantial impact on the overall properties of composite materials. Ash (1.1%), lignin (5–12%), and cellulose (70–82%) make up PALF [29]. PALF have very good mechanical properties in terms of tensile, flexural, and impact strength, which are highly wanted for producing high-quality polymer composites. PALF has the drawback of being difficult to interact with hydrophobic polymers well due to its hydrophilic nature.

The main problem with these natural fibers is that they are hydrophilic, which leads to a weak interfacial connection between the fiber and matrix in polymer composites. It is impossible to use fibers as enforcement materials due to the hydroxyl groups on the fiber surface and several physical flaws [30]. There have been numerous studies conducted in the past on how to modify and change natural fibers to achieve the desired outcomes [12, 31-33]. However, a composite material's mechanical performance is influenced by the kind, orientation, and bonding of the fibers and matrix as well as by both of these factors [34]. These weak fiber connections have a detrimental effect on the mechanical strength of fiber board [30]. The fiber's surface is always treated to control the fiber-matrix adhesion in order to ensure that composites perform well. The interface quality of composites can frequently be improved by applying the proper procedures to the fiber surfaces, such as chemical modification and coupling or compatibility agents that are reacted or deposited onto the fiber. The hydrophilic characteristic of the natural coir fibers was reduced by surface modification using silane [13]. In order to enhance the interface quality of PALFs-polyester composites, In order to enhance the interface quality of PALFs-polyester composites, Mishra et al. [35] showed that the right surface modifications, such as dewaxing, alkali treatment, cyanoethylation, and application of acrylonitrile monomer onto dewaxed PALF, might be employed. In Ref. [36], PALF was treated with different concentrations of NaOH solution (1% w/v, 3% w/v, 5% w/v, and 7% w/v), and the treatment with 5% NaOH led to the largest gain in composite strength when compared to that of untreated fiber.

The single-fiber fragmentation test is one of the most widely used techniques to assess the effects of surface treatments on the composite properties in circular and rigid carbon fibers as well as glass fibers/epoxy resin composites [37, 38]. Recently, the single-fiber fragmentation test has been successfully used to evaluate the interfacial properties of numerous natural fibers reinforced with thermoset materials [39]. Inserting a single fiber into a thin test piece of resin allows for testing. Tensile stress is transferred from the surrounding matrix to the inserted fiber by interfacial shear stress. The applied strain increases until it reaches a level high enough to produce fracture, at which point the fiber repeatedly cracks when its strength is exceeded. Continued application of load causes further fragmentation after the length of the remaining fragment is insufficient for new fractures to form. This situation occurs during the fiber-fragmentation process' saturation stage. The ultimate fragment lengths in a transparent matrix composite can be measured using an optical microscope. The data from the fragmentation test were analyzed using the constant shear lag Kelly-Tyson model to assess the quality of the interface as follows:

$$\tau_a = \frac{r_f \sigma_{ts}}{l_c} \qquad (1)$$

where τ_a is the apparent interfacial shear strength (IFSS), r_f is the fiber radius, and f_{ts} is the fiber tensile strength at a length equal to l_c, the critical fiber length.

In order to apply Equation (1), the critical fiber length, l_c, from the average fiber fragment length, l, must be calculated using Ohsawa's relationship [40]:

$$l_c = \frac{4}{3}\bar{l} \tag{2}$$

The characterization of the interface properties of epoxy resin reinforced with PALF was the focus of this investigation. The reagents employed to treat the PALFs were NaOH and silane. Both the two distinct treated PALFs and the untreated PALFs had their mechanical properties examined. The effect of surface treatments on the interfacial shear strength of PALFs of epoxy resin composites was assessed using a single-fiber fragmentation test. A common data reduction technique was used to assess the test data for fragmentation. The surfaces of treated and untreated PALFs were characterized using a scanning electron microscope (SEM).

EXPERIMENTAL

Ingredients

The pineapple leaf fibers used in the current investigation were meticulously taken off the leaves of the pineapple plant and thoroughly cleaned with distilled water to remove dirt, filth, and other surface pollutants. After that, they spent 48 hours at 70°C in the oven. The two compounds used in this investigation were 3-aminopropyltriethoxysilane (3-APS) and NaOH (sodium hydroxide 5% w/v soln, R&M) (Sigma Aldrich, Jasa Sejiwa Enterprise). The epoxy resin of choice was Epikote 828 (Hexion Specialty Chemicals, USA), a diglycidyl ether of Bisphenol A-type epoxy. Tetraethylenetriamine (TETA) from the Yuka-Shell Corporation was used as a hardener. Both acetic acid and ethanol were AR-grade and used exactly as received.

METHODS

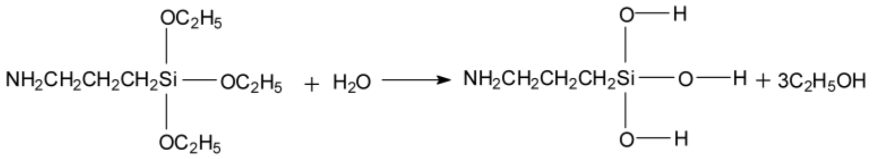
Surface Modification of Fibers

Extraction and Alkali Treatment of PALF

The pineapple plant's mature harvested leaves were submerged in water for four weeks to make them pliable for simple fiber separation. The hemicellulose cement matrix was broken down by fungi and bacteria as a result of this process, which also liberated the fiber bundle and improved fiber separation. The separated fibers were cleaned in distilled water and dried in an oven at 70°C. Clean and dried PALF fibers were soaked in a sodium hydroxide solution (5% w/v) for two hours at room temperature. The fibers were then rinsed with distilled water until the NaOH was eliminated, or until the rinse water showed no evidence of alkalinity. This was done by first washing the fibers with distilled water mixed with a few drops of acetic acid, then repeating the process with distilled water. The fiber was dried by air for two days after washing. After being air dried for two days, the fibers were then placed in an oven set to 80°C for 6 hours. The NaOH treatment enhances the fiber surface's adhesive properties by giving it a roughened topography and removing both natural and artificial contaminants.

Silane Treatment

In Ref. [31], coir fibers were treated with 3-aminopropyl trimethoxy silane and tetramethoxy orthosilicate after the coir fiber underwent an alkali pretreatment that improved all of the mechanical properties and water absorption of the resulting composites compared to that of untreated fiber composite. According to the literature, 5-weight-percent silane (3-APS) was hydrolyzed using a solution of water and ethanol (40:60 w/w) (weight percentage regarding the fiber) [41, 42]. The solution was stirred continuously for an hour after the pH was reduced to 4 with acetic acid. The fibers were soaked in this solution for three hours before being removed. After cleaning, the fibers were left to air dry for three days. After that, the fibers were baked for 12 hours at 80°C to finish drying. When there is water present, the ethoxy groups on the silane coupling agents hydrolyze and produce silanol groups [41, 42]. The hydrolysis of three ethoxy groups in APS yields three hydroxyl groups. A stable covalent bond is formed with the cell wall as a result of the reaction between the silanol and OH groups on PALF, and this bond is chemisorbed onto the fiber surface. The silanols can also create siloxane connections with the matrix polymer, according to Dupraz et al. [41]. The silanes act as molecules that link the matrix and the fiber as a result.



$$NH_2CH_2CH_2CH_2Si(OC_2H_5)_3 + H_2O \longrightarrow NH_2CH_2CH_2CH_2Si(OH)_3 + 3C_2H_5OH$$

Aminopropyltriethoxysilane (3-APS) **Silanol**

$$\textbf{(Silanol)}\ NH_2CH_2CH_2CH_2Si(OH)_3 + \textbf{(Water)}\ H_2O + \textbf{Fiber-}OH \longrightarrow NH_2CH_2CH_2CH_2Si(OH)_2O\textbf{-Fiber}$$

Alkali and Silane Treatment

The fibers were immersed into distilled water with combination of sodium hydroxide solution (5% w/v) and 5 wt% silane (NaOH-silane) for 3 h. The fibers were thoroughly washed with running water after treatments a number of times until pH levels were balanced. The fibers were then dried for 48 hours in an oven at 80°C.

CHARACTERIZATIONS

Morphological Observation

The changes in fiber surface topography after surface treatments were assessed using a LEO145 VP SEM scanning electron microscope. Prior to SEM analysis, the samples were gold-coated for a minute using a gold coating sputter.

Diameter Measurement

A single fiber was tested for diameter at room temperature. Natural fibers are particularly challenging to accurately determine the diameter of due to their irregular form and unequal thickness. A natural single fiber bundle's matrix is largely made up of lignin, hemicelluloses, and element fibers. Therefore, the cross section of a single fiber bundle is not circular. Circular cross section was assumed in the computation of tensile properties even though the cross section was irregular along the length of the fiber. We measured the diameters of untreated and treated single fiber bundles using an image analyzer (Figure 1). Five times each sample was measured at three distinct locations along its length to determine the average diameter of each fiber.

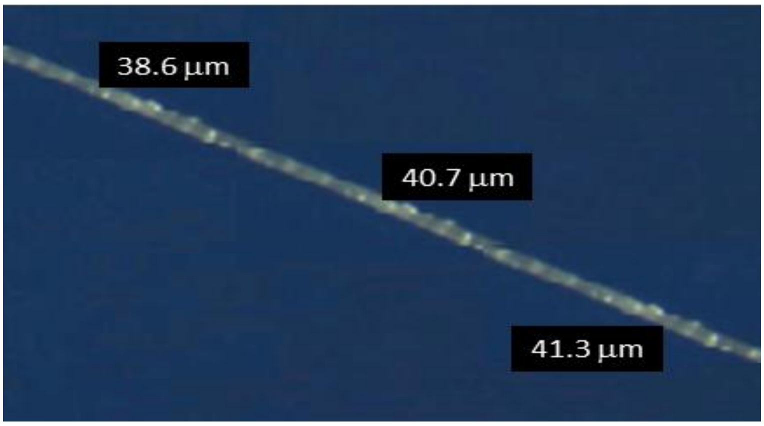


Figure 1: Measuring the diameter of the fiber

Single Fiber Tensile Test

The single fiber test was carried out according to the steps outlined by Cheng et al. [37]. Hand-selected at random from the untreated, NaOH, and epoxy treated PALFs, single fibers were placed onto light, thin window cards with a 4.5 mm diameter hole punched in them as shown in Figure 2(a). The fiber was secured in place by folding the opposing cards and sticking them together with adhesive. Fiber misalignment was present in the middle of the holes in the samples that failed this test. The samples were then pulled in uniaxial tension using a 10 N load cell on the little tester Lloyd LRX-Plus at a displacement rate of 0.52 mm/min. Figure 2(b) illustrates how the supporting material for the fiber was severed before the test. When the specimens were tested, only data for the fibers that failed under tension were recorded, and the testing was closely supervised. In a typical test, the force progressively decreases to zero before the tension rapidly decreases to the point where the fiber shattered. In rare cases, the fibers could peel away from the supports because of insufficient adhesion, which caused them to slide inside the windows. Since the load did not quickly reduce to zero in these circumstances at the fracture condition, these facts were not taken into consideration when computing the fiber strength. 30 to 40 samples were used to evaluate it. The weibull statistical approach was used to calculate the values of the weibull modulus after averaging the results of the tensile tests (m) [37]. To compare the strength of treated and untreated PALFs at the critical length, l_c [37], the following relationships were used.

$$\sigma_{ts} = \bar{\sigma}\left(\frac{l}{l_c}\right)^{\frac{1}{m}} \qquad (3)$$

where $\bar{\sigma}$ the average tensile strength of the fibers, and l is the fiber length chosen for the strength measurements. For the untreated and treated PALFs, l = 4.5 mm.

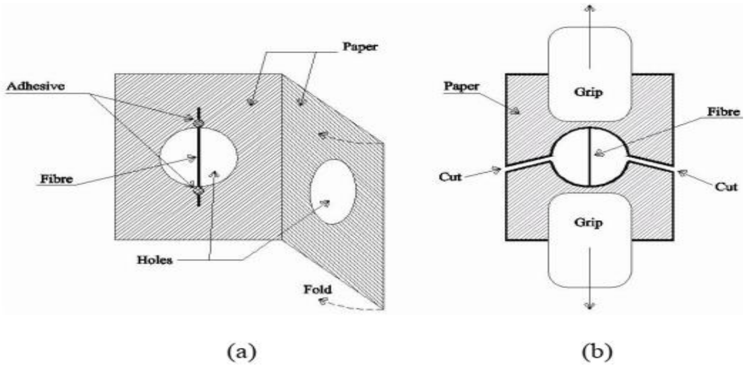


Figure 2: Schematic diagram of the preparation of a single fiber strength test specimen (a) Preparation and (b) testing of single fiber strength test specimen

Single-Fiber Fragmentation Test

Test specimens were made ready for single-fiber fragmentation using the method outlined by Cheng et al., [37]. A specimen with a single fiber implanted longitudinally in a resin matrix is utilized to perform the fragmentation test, and this specimen has a strain to failure value that is two to three times higher than fibers extracted from pineapple leaves. The testing was carried out on an Instron universal testing machine with a cross-head speed of 0.50 mm/min. The samples were stretched to 15% applied strain to completely saturate the fragmentation process. The fiber fragment lengths were determined in transmitted light using an Olympus light microscope with a graded eyepiece. The interfacial failure mechanisms that took place at the site of the fiber fractures were examined using a transmitted polarized light microscope and a Nikon DN100 Digital Net Camera. Using the Kelly-Tyson model provided in Equation, the determination of τ_a was accomplished (1).

OUTCOMES AND DISCUSSION

Scanning Electron Microscopy (SEM) Observation

SEM analysis was employed to look at the surface morphology of PALF. Interfacial bonding between fibers and resin results in better mechanical characteristics and is significantly impacted by fiber surface [43]. Identification of the structural abnormalities caused by the treatment that assist in cleaning and smoothing fibers depends on the microscopic analysis of fiber surface morphology. Figure 3 displays the PALF surfaces before and after treatment. Figure 3a shows the SEM micrograph of PALF that has not been treated. Untreated fibers have a surface that is covered in a number of impure chemicals. The fiber after being treated with NaOH, which improved surface quality and made the surface of the fiber very smooth, is shown in Figure 3b. The lack of any foreign substance, as well as the presence of lignin and hemicellulose, is indicated by small surface pores and a smooth surface. The pores may help improve interfacial bonding [44]. Figure 3c, a fiber that has been silane-treated, shows a

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surface that is clear and uncontaminated. With considerable effectiveness, silane treatment can be used to eliminate lignin and hemicelluloses from natural fibers. Poor bonding between the fibers and matrix is the result of the NaOH-silane treatment's minimal impact on the morphology of the fibers in Figure 3d [45]. Silane-treated fibers are smoother and more able to bond with the matrix, whereas NaOH-treated fibers can clean up surface impurities, according to Puglia et al., [46].

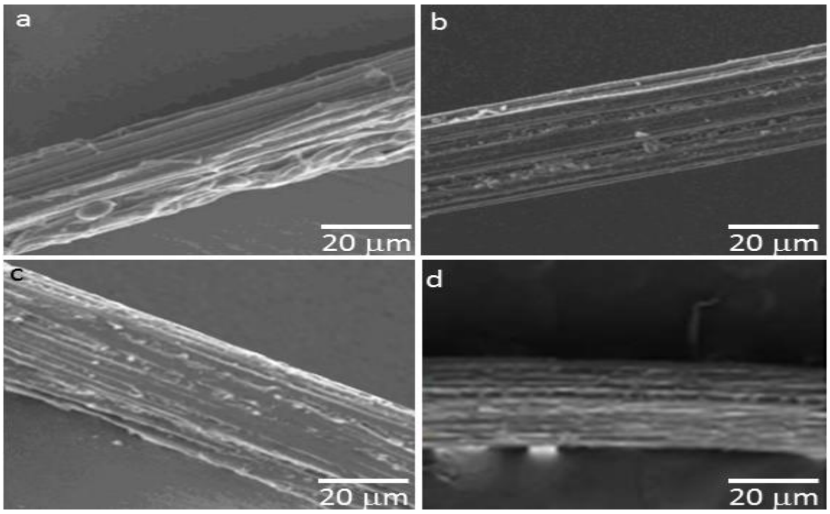

Figure 3: SEM of (a) untreated, (b) NaOH, (c) silane and (d) NaOH-silane treated of PALF

Diameter Measurement

There was a small percentage of chemically treated fibers, and these fibers had a rougher structure and more surface area to promote better interfacial adhesion [47]. The diameters of treated and untreated PALF are shown in Table 1. The difference in diameter between treated and untreated fibers is clearly shown in Figure 4. These chemicals targeted both of the fibers' surfaces and damaged the lignin and hemicellulose web, detaching the fibers from the bundles [48]. Chemically treated fibers had smaller, more consistent diameters than untreated fibers in this study. The 5% silane treatment and the 5% NaOH treatment both had a better impact on both fibers and were the most effective of all the treatments. All treated fibers have smaller diameters than untreated fibers, with some minor variances seen in Table 1's comparison of the effects of various treatments on fiber diameters. Fibers may become weaker and more fragile with longer soaking times and greater NaOH concentrations [49]. The results of this study conclusively show that fiber treated with 5% silane was able to separate the lignin and hemicellulose web of fiber bundle while maintaining its mechanical properties, as shown in Table 1.

Table 1: Diameter, tensile strength, tensile modulus, and IFSS of PALF

Testing	Untreated	NaOH-silane	NaOH	Silane
Fiber diameter (µm)	75.5	52.1	48.3	41.2
Tensile strength (MPa)	293.5	420.3	428.2	625.8
Tensile modulus (MPa)	5230.8	6371.3	8432.4	10189.8
IFSS (MPa)	1.65	1.98	1.75	2.42

Mechanical Features

Tensile Strength

The difference in tensile strength between untreated and treated PALF is seen in Figure 5. Chemical treatments improved the tensile strength of natural fibers [50]. The elimination of contaminants using alkali treatment has also been very successful [51], and the tensile strength can be impacted by the time and concentration of NaOH used in the process. Furthermore, it was shown that silane and NaOH treatments improved the tensile strength of the fibers in comparison to untreated fibers. Chemical processing of fibers increased their tensile properties by removing lignin and hemicelluloses and cementing the fibers together with cellulose. Figure 5 shows how chemical treatments affect the tensile strength of PALF. Chemically treated fibers that contained 5% silane were used to demonstrate the highest tensile strengths. Untreated PALF had a tensile strength of 293.5 MPa. The tensile strength of PALF with NaOH-silane treatment was the lowest of all the treated fibers. PALF fibers treated with 5% NaOH concentration showed comparable strength to that of PALF fibers treated with NaOH-silane. Fibers were treated with 5% NaOH to easily remove surface impurities on PALF. However, fiber that had been silane treated showed better interfacial adhesion between matrix and fibers because lignin and hemicellulose were reduced. Higher chemical concentrations might be more efficient in removing impurities, but they might also have a negative impact on tensile strength because they might rupture fiber surfaces and degrade lignocellulose [52]. Compared to other therapies, silane therapy showed a much stronger effect. In the case of PALF, only the silane treatment is significant, and other treatments have a very similar tendency to be ineffective.

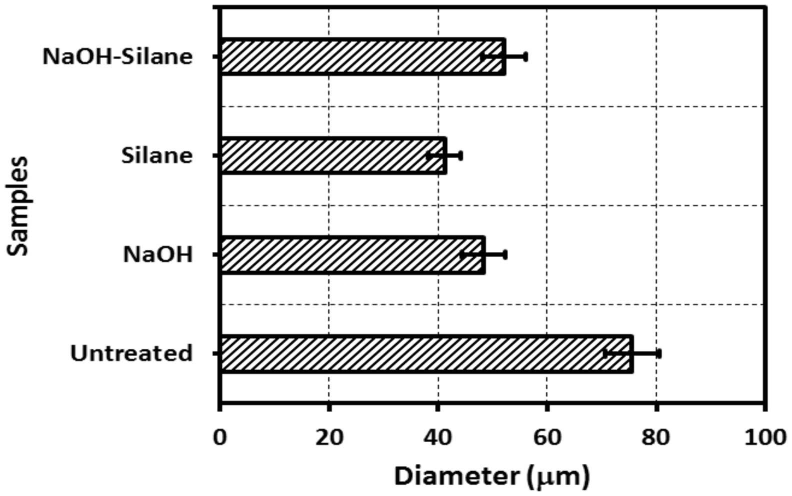

Figure 4: Diameter of PALF

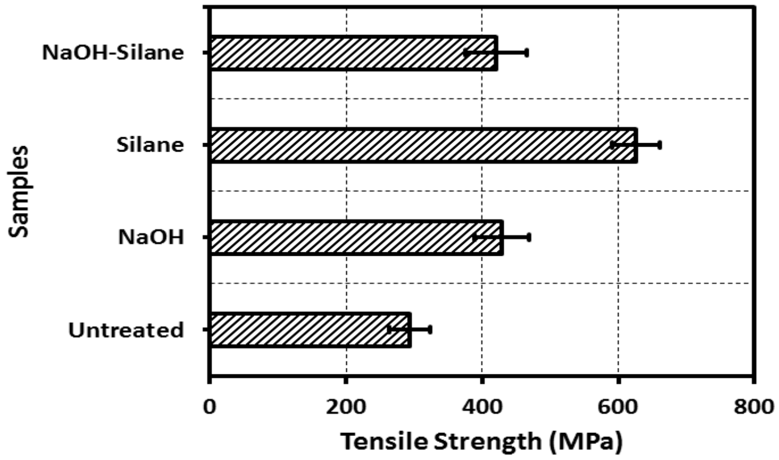

Figure 5: Tensile strength of PALF

Tensile Modulus

The stiffness of a material can be calculated using the tensile modulus measurement. The chemical treatments increased the tensile moduli of PALF, as seen in Figure 6. In terms of their tensile moduli, treated and untreated fibers have varying tensile strengths. The tensile moduli of the two NaOH-silane-treated fibers were higher than those of the untreated fibers. The tensile moduli increased to their highest level after the fibers were treated with a 5% silane solution. All tensile moduli are shown in Table 1. The silane-treated PALF has a 95%

greater tensile modulus than the untreated PALF. In comparison to untreated PALF, the tensile moduli of PALF that had undergone chemical treatment with NaOH at a 5% concentration were also larger. PALF treated with NaOH, however, displayed 17% less than PALF treated with silane. Low tensile moduli of fibers treated with NaOH may cause fiber breakdown as a result of severe delignification [53]. All surface contaminants were removed thanks to the silane-treated fiber's high tensile modulus, which was also visible in SEM. While fiber that had been treated with NaOH-silane had a relatively low tensile modulus, only silane had the highest maximum tensile strength and modulus. The tensile modulus of NaOH-silane fiber significantly dropped as a result of a greater solution concentration.

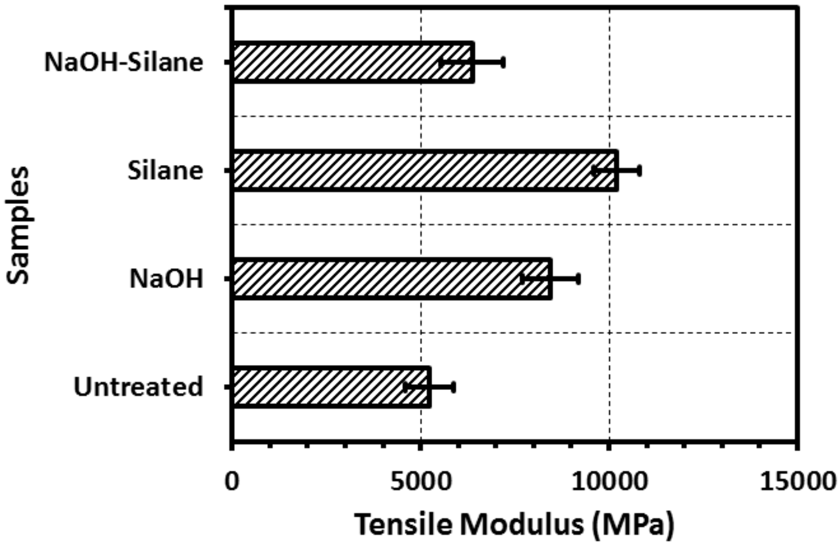

Figure 6: Tensile modulus of PALF

Single-Fiber Fragmentation Test

The interfacial shear strength test is used to determine the interfacial properties of fiber-reinforced polymer composites (IFSS). The interfacial bond between the matrix and the fibers is shown in Figure 7. The silane-treated fibers had the highest IFSS, as the results of the fragmentation tests clearly showed. The fiber with the little matrix drop was quite obvious in Figure 7a. Following the test, Figure 7b of the same fiber displayed the matrix break clearly. IFSS values for untreated PALF were 1.65 MPa (Table 1). In comparison to untreated PALF fibers, silane-treated PALF fibers have an IFSS that is about 47% higher. The strength of PALF treated with NaOH only increased by 6%. The IFSS of PALF was only increased by 20% in fibers treated with NaOH-silane. Silane-treated fibers perform better in terms of IFSS of PALF compared to NaOH- or NaOH-silane-treated fibers. The fibers that had been silane-treated had the biggest gains in IFSS. The elimination of lignin and hemicellulose was most likely the cause of this result. After these chemicals were removed, the coupling agent infiltrated the fibers and was deposited in the interfibrillar regions, helping to form an anchor between the matrix and the fibers. Effects of fiber treatments on composites constructed of unsaturated polyester and reinforced with hemp fiber in terms of interfacial shear strength [54].

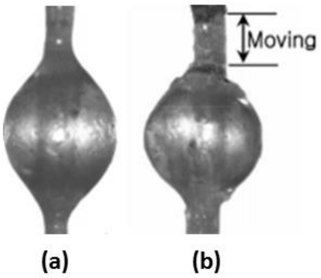

Figure 7: Single fiber microbonding test (a) before test; (b) after test

CONCLUSION

The lack of compatibility between natural fibers and the matrix is the fundamental drawback of using them in thermosets. Therefore, to modify the characteristics of PALF fibers, surface treatments were employed. For the modification of PALFs, two chemical reagents, NaOH and a silane, were employed. This study looked at how treatments with

NaOH, silane, and NaOH-silane affected the morphological, structural, and tensile characteristics of PALF. The SEM graphs demonstrate the removal of non-cellulosic components from the silane-treated PALF. By using PALF, the hydrophilic characteristics are reduced. Due to the removal of the contaminants, silane-treated fibers exhibit a decrease in diameter when compared to untreated PALF. In comparison to untreated, NaOH-, and NaOH-silane-treated fibers, the PALFs' tensile modulus and strength were boosted by the silane treatment. The interfacial shear strength proportional to the degree of interfacial adhesion between PALF and epoxy resin matrix was examined using the single fiber fragmentation test. When compared to the untreated fibers, the PALFs' NaOH and silane treatments improved the IFSS of epoxy composites by 6% and 47%, respectively, and improved the effectiveness of stress transfer at the interface of composites. These findings imply that silane and NaOH surface treatments significantly improve interface quality and boost the durability of PALFs. To ascertain the impact of surface treated PALFs on the unidirectional PALFs-epoxy composites, future research must be done.

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